

Determination of the Effect of the Addition of Pumice on the Technological Properties of Wall Tile Using the Factorial Design Method

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Abstract

In this study, the effects of the addition of pumice which has alkali and silica rich content on wall tile product properties were investigated using a factorial experimental design. A 2²×3¹ mixed-level factorial experimental design was used to determine the effect of three main factors and interactions between them on wall tile product characteristics. Instead of pegmatite, ground pumice was added at different amounts to the standard wall tiles recipe and five compositions were prepared. Factors affecting the properties of the product were selected as pumice grinding time (10 and 15 minutes), the amount of pumice (0%, 7% and 14%) and firing temperature (1100°C, 1150°C). Linear shrinkage, water absorption, bulk density, total porosity and flexural strength values of samples were measured and the microstructures were investigated. As the amounts of pumice were increased, the amount of the glassy phase was escalated while the total porosity and water absorption values were decreased, bulk density values was increased. Pumice addition instead of pegmatite caused the dissolution of anorthite crystals in the microstructure, facilitating a decline in the amount of anorthite crystals with its high alkaline content and flexural strength values decreased accordingly. The grinding time and the amount of pumice were found to be active on examined features such as the main factors and also the interaction factor.

Keywords

Experimental Design; Wall Tile; Pumice; Microstructure

Introduction

Wall tiles have high porosity and higher water absorption than other ceramic tiles. Their water absorption is higher than 10% according to European standards (EN 14411). While feldspars and alkali containing raw materials are sometimes used, carbonaceous raw materials (clay, calcite and so on) are generally used in wall tiles [web site 1]. The typical wall tile formulations are generally contain clays, silica,

fillers (feldspathic sands) and carbonates [Garcia et al., 2008]. The chemical composition of industrial wall tile bodies is 55-70% SiO₂, 12-20% Al₂O₃, 2-12% (CaO+MgO) wt.% [Sausa and Holanda, 2005]. Each component within the formulations affects the final properties differently. The calcareous content in carbonates (chalk, limestone, marble and dolomite) improves the densification behavior, the shrinkage on firing and sintering temperatures of bodies. Furthermore they develop new crystal phases with quartz, alumina during firing and those crystals are responsible for mechanical strength. Also optimum combinations of various clays (ball, kaolinitic, bentonitic) provide plasticity and green strength [Das et al., 2005]. There has been limited research in the literature on the use of different raw materials in wall tiles. Pumice is one of these raw materials.

Although pumice can be found in many parts of the world, Turkey has the richest and produces the highest quality pumice deposits. Pumice from Turkey, with a high color quality and texture, has a large number of foreign markets [Civan, 2011]. Pumice is a porous, spongy, glassy mineral, resulting from past volcanic activity and is resistant to physical and chemical effects [Cayırlı, 2008]. In terms of chemical composition, it contains 65-70% SiO₂, 13-15% Al₂O₃, 7-8% Na₂O-K₂O, 1-3% Fe₂O₃, 1-2% CaO, 1-2% MgO and low amounts of TiO₂ [web site 2]. The physical and chemical structure of this mineral is commonly used in construction, agriculture, textile, chemical, cosmetics and many other industries including glass manufacture [Poyraz et al., 2006; Deniz and Onur, 2002]. Pumice is used in light weight concrete production due to its low unit weight [Demirel and Ekici, 2008]. Considering this mineral as a ceramic raw material allied to the construction sector will

contribute to the country's economy.

Experimental design techniques have offered guidance in overcoming problems such as optimization, active factor determining, and mixture designing. Experimental design is the most effective and economical method for determining the process variables' relationships with each other and their critical levels [Rocak et al., 2002]. One of the experimental design methods is factorial design. In the factorial experimental design for the whole trial or repeated trials, all the combinations of the examined factors have been investigated [Montgomery, 2001]. Assessing the factors affecting the ceramic production process, many studies on experimental design methods are documented in the literature. Another study comprises the determination of the effect of pressing pressure, firing temperature and time on linear shrinkage, water absorption, fracture toughness and densification using a two-level full factorial design in the production of porcelain tiles [Bondioli et al., 2007]. As a result of such experiments, it was determined that the most effective main factors were sintering temperature and pressing pressure, and that the most effective interaction factor was sintering temperature*time for controlling the process of densification. With a limited amount of tests on shrinkage, defining the effect of various factors was possible. Zauberas and Boschi (2004) examined the effects of pressing pressure, maximum firing temperature and holding time factors on linear shrinkage, water absorption, flexural strength and pyroplastic deformation in the production of four different porcelain tile compositions using a 2^k factorial design. The most appropriate factors and their interactions to be changed were determined to characterize the compositions and to achieve the desired objective [Zauberas and Boschi, 2004]. In another study, statistical techniques were used to determine the effect of process variables on cordierite formation from the mechanochemical synthesizing of kaolin, talc and $\text{Al}(\text{OH})_3$ mixture. As a result of these techniques, it was determined that the most important main factor that affects the synthesized product is temperature and that the most effective interaction factor is the addition of Mg borate*grinding time [Yalamac and Akkut, 2006]. Kićević et al. (1996) examined the relationship between the mechanical properties of sintered fused silica and cristobalite content by a multifactorial experimental design, and analyzed the relationship between bending-compression strength and cristobalite content by the

response surface method. 6-8% cristobalite content by volume was found to improve the mechanical properties of the material [Kićević et al., 1996].

In this study, Nevsehir (Turkey) region's acidic pumice was added by a gradual reduction of pegmatite in the wall tile recipe. Linear shrinkage (%), bulk density, water absorption (%), total porosity (%) and flexural strength changes were analyzed using the factorial design.

Materials and Methods

Acidic pumice obtained from the Nevsehir Denge Bims Pumice Plant was used in the experiments. The pumice was ground in a ring mill for 10 and 15 minutes, and then added to recipes. The chemical composition of raw materials used in the recipe of wall tile and pumice are given in Table 1. In this study, instead of pegmatite, pumice usage thanks to its melting properties and high silica content was discussed. The amounts of raw materials that were modified in the recipes are given in Table 2. The particle size distributions of the recipes subjected to wet-grinding were measured using a Malvern Mastersizer 2000 G particle size device and the particle size distributions of the recipes are given in Figs 1-5. The values of d_{50} and d_{90} are similar for pumice added and pumice free slurries at particle size distribution graphics. While recipe 3 which contains 15 minute ground pumice, has a d_{90} value of about 20 μm similar to the other recipes, the same recipe which contains 10 minute ground pumice has a d_{90} value of about 30 μm . Even with a 5 minutes difference in the grinding of the pumice, it is effective in particle size distribution. The slips were dried, and then humidified with 6% wt. water. The humidified mixtures were shaped by the uniaxial pressing technique in a 50 mm x 100 mm x 8 mm dimension mould at 450 kg/cm². The factors of mixed-level factorial design experiments (grinding time of pumice, amount of pumice and firing temperature) and their levels are given in Table 3. The examined main factors and interaction factors expressed using the main factors are given in Table 4. two different pumice grinding period was applied (10 min., 15 min.), the amount of pumice has 3 amounts (0%, 7%, 14%) and the firing temperature has 2 settings (1100°C, 1150°C), respectively. The experiments were repeated twice and the results were analyzed using a Minitab 14 software program. Linear shrinkage, bulk density, water absorption, porosity and the flexural strength of sintered tiles at industrial conditions were inspected.

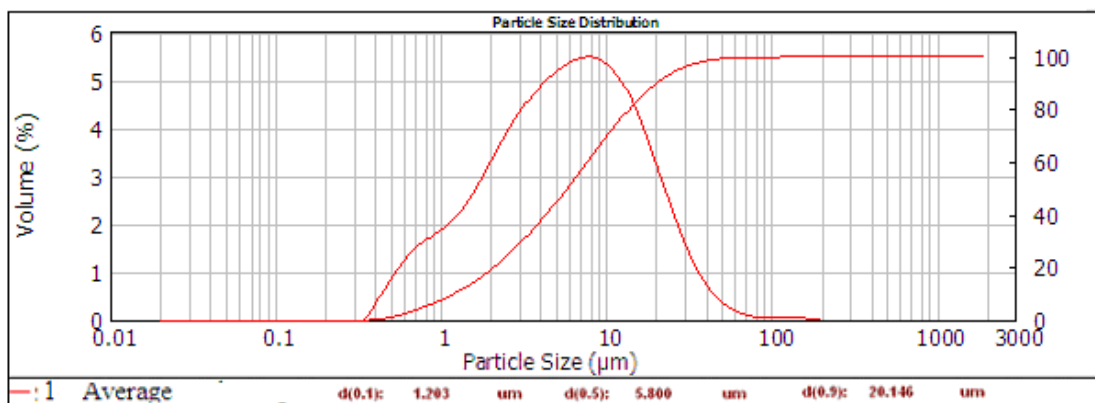


FIG. 1 PARTICLE SIZE DISTRIBUTION OF RECIPE 1.

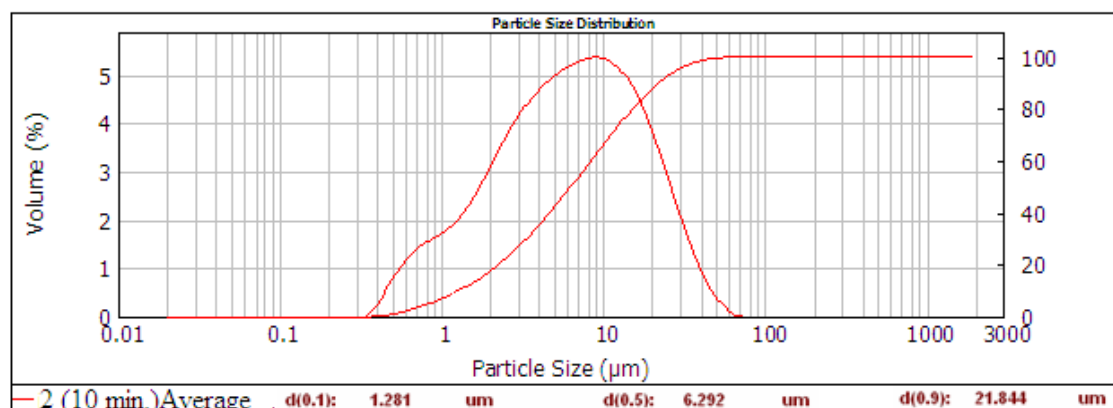


FIG. 2 PARTICLE SIZE DISTRIBUTION OF 10 MIN. GROUND PUMICE CONTAINING RECIPE 2.

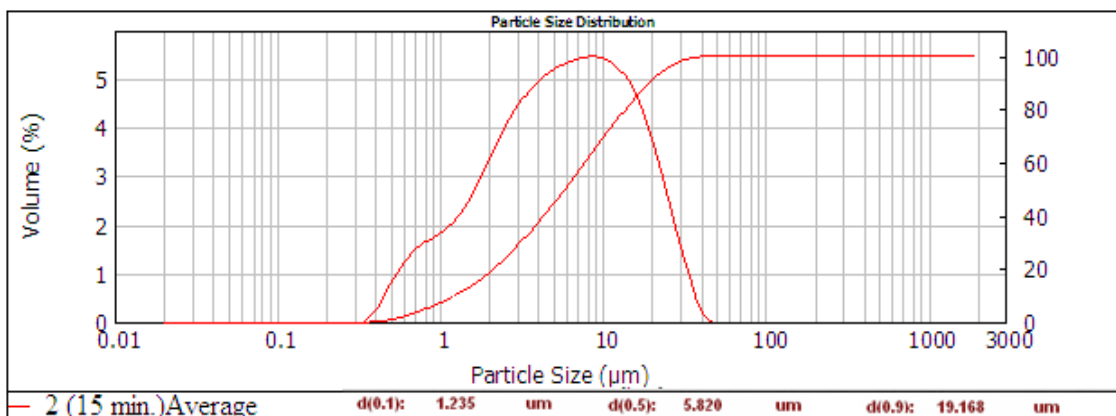


FIG. 3 PARTICLE SIZE DISTRIBUTION OF 15 MIN. GROUND PUMICE CONTAINING RECIPE 2.

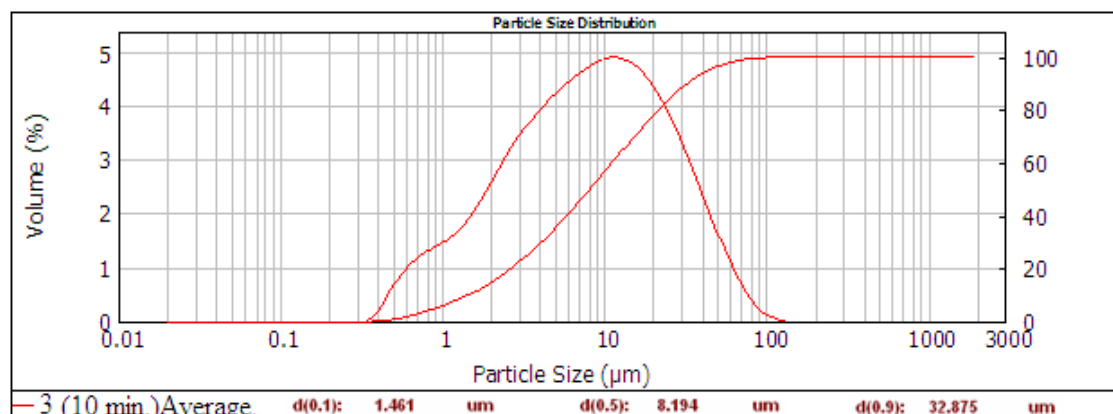


FIG. 4 PARTICLE SIZE DISTRIBUTION OF 10 MIN. GROUND PUMICE CONTAINING RECIPE 3.

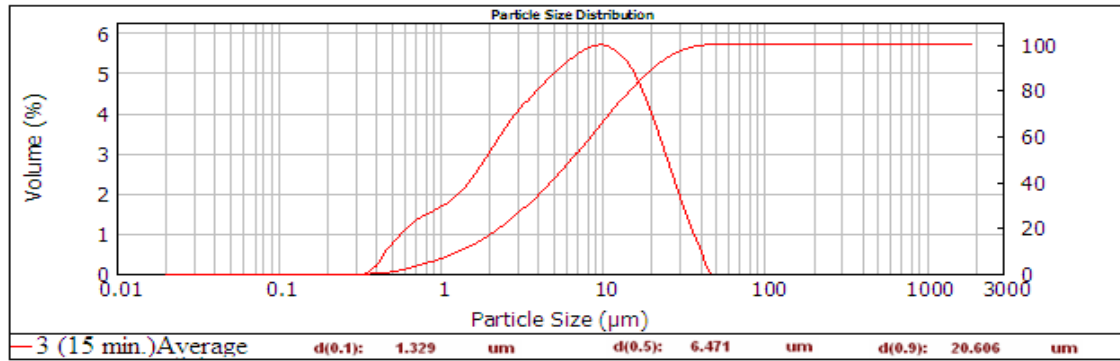


FIG. 5 PARTICLE SIZE DISTRIBUTION OF 15 MIN. GROUND PUMICE CONTAINING RECIPE 3.

TABLE 1 CHEMICAL ANALYSIS RESULTS OF RAW MATERIALS (WT.%)

Raw materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	Cr ₂ O ₃	TiO ₂	SO ₃	L.I.*
Clay	61.04	26.47	0.44	0.23	-	0.28	0.16	0.17	0.09	0.74	0.27	10.04
Kaolin	63.21	21.88	3.19	0.14	0.62	-	2.46	0.04	-	1.28	0.08	7.05
Pegmatite	66.48	20.04	1.32	0.50	0.38	2.68	2.78	0.06	-	0.96	0.05	4.69
Calcite	0.33	-	0.22	56.32	-	-	-	0.02	0.07	-	-	43.02
Silica Sand	91.58	5.07	0.51	0.07	0.04	0.10	0.52	-	-	0.24	-	1.82
Pumice	73.18	12.43	1.22	0.82	0.03	4.06	4.62	-	-	0.08	-	3.53

*L.I.: Loss of ignition

TABLE 2 AMOUNT OF MODIFIED RAW MATERIALS IN RECIPES

Recipes	1	2	3
Clay	35	35	35
Kaolin	35	35	35
Pegmatite	14	7	-
Silica Sand	10	10	10
Pumice	-	7	14

TABLE 3 EXAMINED FACTORS AND THEIR LEVELS

Factors	Levels		
	1	2	3
Grinding time of pumice (minutes)	10	15	-
Amount of pumice (wt.%)	0	7	14
Firing temperature (°C)	1100	1150	-

TABLE 4 EXAMINED MAIN FACTORS AND INTERACTION FACTORS

Main Factors	Interaction factors	
Grinding time of pumice (min.)	Grinding time of pumice* amount of pumice	Grinding time of pumice*the amount of pumice*firing temperature
The amount of pumice (wt.%)	Grinding time of pumice* firing temperature	
Firing temperature (°C)	The amount of pumice*firing temperature	

Shrinkage is generally expressed as a percentage and may concern the linear, area or volume shrinkage. The choice of measure depends on practical considerations [Singer and Singer, 1963]. The linear shrinkage of tiles was determined by $\Delta L/L_0$.

The bulk densities and water absorption (%) of the sintered samples were calculated by the water immersion Archimedes principle using the related equations (Eqs. (1) and (2)):

$$\text{Bulk density} = \frac{w_d}{w_w - w_s} \times \rho_{H_2O} \quad (1)$$

$$\text{Water absorption}(\%) = \frac{w_w - w_d}{w_d} \times 100 \quad (2)$$

where w_d is the dry weight of the sample, w_w is the wet weight of the sample and w_s is the weight of the solid suspended in water.

For determination of the total porosity by Helium pycnometry (Eq. 3), the bulk density used is measured by the Archimedes method and the theoretical density values obtained from Helium pycnometry.

$$P(\%) = \frac{(\rho_t - \rho_b)}{\rho_b} \times 100 \quad (3)$$

In the equation, $P(\%)$ is total porosity (%), ρ_t is theoretical density (g/cm^3) and ρ_b is bulk density (g/cm^3) [Andreola et al., 2000].

Flexural strength of tiles was measured for each tile using the three-point bending test (Gabrielli CR5). The strength is defined as the maximum tensile stress at failure and for a rectangular test specimen can be calculated using the general flexure stress formula:

$$S = \frac{Mc}{I} = \frac{3PL}{2bd^2} \quad (4)$$

where M is the moment, c is the distance from the neutral axis to the tensile surface, and I is the moment of inertia. For a rectangular test specimen and three-point loading P is the load required to cause failure, $M = (L/2)(P/2)$, $I = bd^3/12$ and $c = d/2$, where d is the thickness of the specimen and b is the width [Richerson, 1992].

X-ray diffraction (XRD) was used for a qualitative determination of the crystalline phases in the fired formulations. This was performed using a Rigaku Rint 2200 diffractometer (with $\text{CuK}\alpha$ -radiation) at 40 kV and 30 mA. Samples were scanned from 2α , 5 to 70° , at a scanning speed of $2^\circ/\text{min}$. Also, a Rietveld Analysis was used for quantitative amounts of all the phases present in the fired formulations. For SEM observations, a Zeiss Evo 50 EP scanning electron microscope (SEM) was used in secondary electron (SE) imaging mode. Chemical etching of polished surfaces of the samples was achieved by dipping the selected samples in a 5% hydrofluoric acid (HF) solution at room temperature for 40 s before examination under SEM.

Results and Discussion

The linear shrinkage, water absorption, bulk density, porosity and flexural strength values of 24 samples which were prepared according to $2^2 \times 3^1$ mixed-level factorial design with two replications were measured. Factors and their interactions were analyzed with an ANOVA table for these properties of the samples. An ANOVA table provides an understanding of whether or not there is a difference among the factor levels and to be able to determine whether any interactions are meaningful [Montgomery, 2001]. In the ANOVA table which includes the effective main factors and their interactions for bulk density in Table 5, DF represents degree of freedom, Seq SS represents the sum of squares, MS represents mean square, and the F distribution is used to determine the differences between factor variances in the ANOVA table. So, the

largest F value is the most effective factor in the model. P values indicate the ratio of the unadmitted region (P values which are lower than the admitted α are effective). $\alpha = 0.05$ value is used (with a 95% confidence interval). Main factors and their interactions with meaningful effectiveness are added to the ANOVA table error term [Montgomery, 2001]. If the P value of the main effect and the interactions of the factor is higher than 0.05, this interactions and main effect are added to the error table. The firing temperature has a 48.1% effect on linear shrinkage. The interaction graphs of effective factors are given in Fig. 6 for linear shrinkage. An addition of pumice amount up to 7% reduces the linear shrinkage of the samples, but the linear shrinkage increases with a 14% rise in the amount of pumice. This is from the high total alkali content of pumice which we used in place of pegmatite. Vitrification of ceramic bodies early starts with alkali and earth alkali oxides [Aras and Demirhan, 2004; Kocabas, 2007]. Therefore, by increasing the amount of pumice and firing temperature, the linear shrinkage of samples reached higher values.

TABLE 5 ANOVA TABLE OF LINEAR SHRINKAGE FOR EFFECTIVE FACTORS

Factors	DF	SeqSS	MS	F	P
Grinding time of pumice (min.)	1	0.23602	0.23602	37.12	0.000
The amount of pumice (wt.%)	2	0.39403	0.19702	30.99	0.000
Firing temperature ($^\circ\text{C}$)	1	0.72802	0.72802	114.51	0.000
Grinding time of pumice* the amount of pumice	2	0.04603	0.02302	3.62	0.049
Error	17	0.10808	0.00636		
Total	23	1.51218	-		

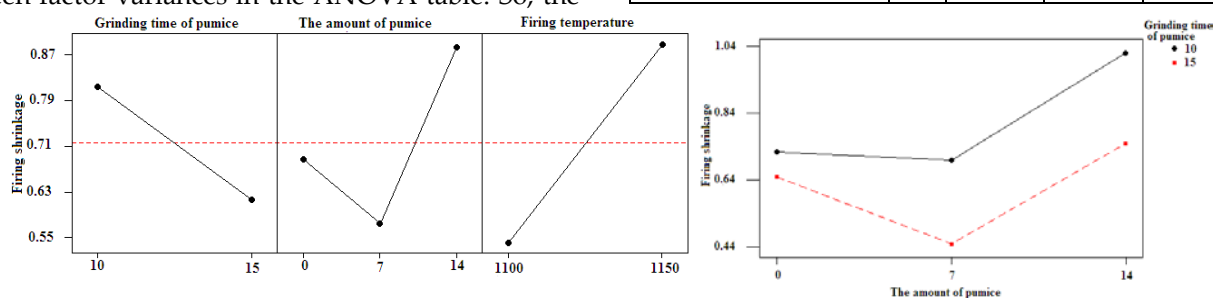


FIG. 6 MAIN EFFECTS AND INTERACTION EFFECT PLOTS FOR LINEAR SHRINKAGE.

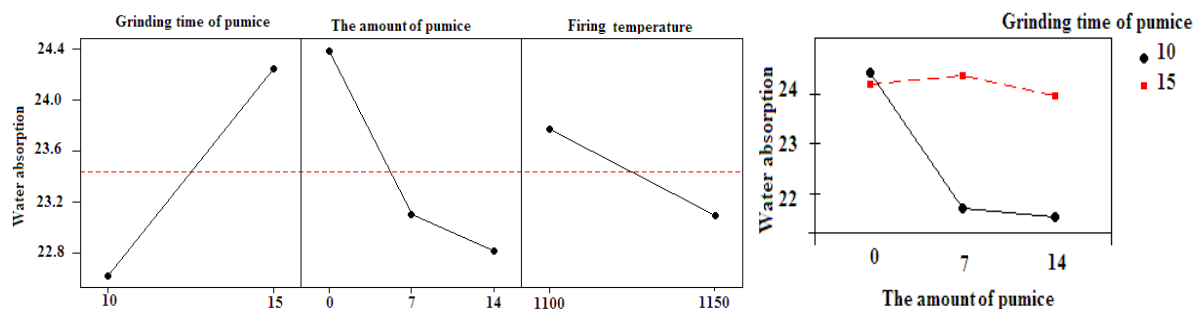


FIG. 7 MAIN EFFECTS AND INTERACTION EFFECT PLOTS FOR WATER ABSORPTION.

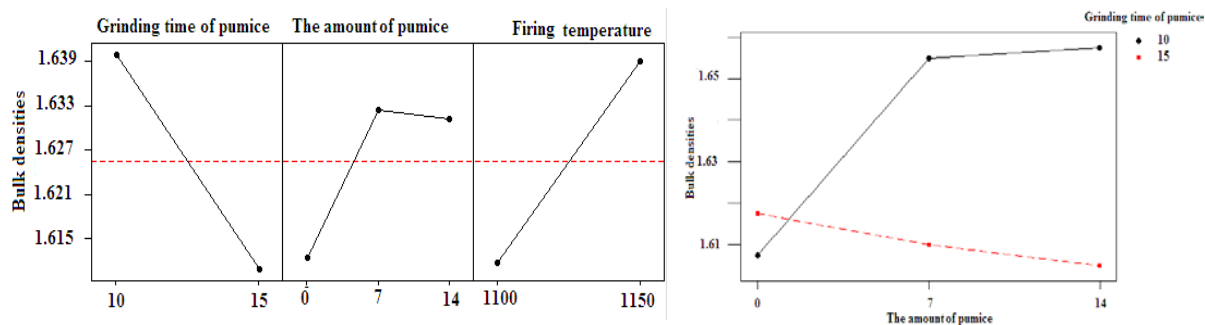


FIG. 8 MAIN EFFECTS AND INTERACTION EFFECT PLOTS FOR BULK DENSITY.

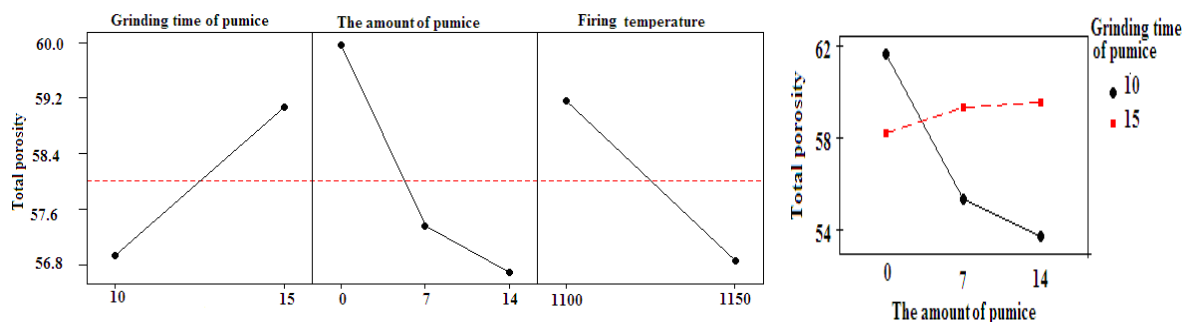


FIG. 9 MAIN EFFECTS AND INTERACTION EFFECT PLOTS FOR TOTAL POROSITY.

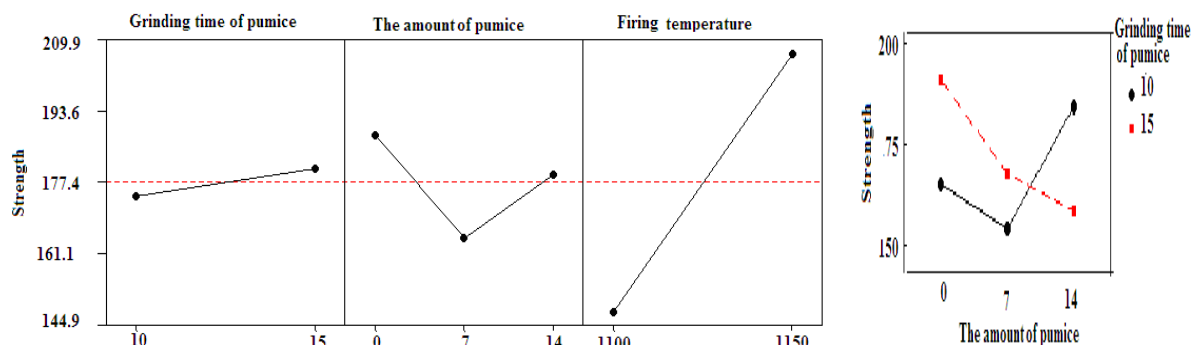


FIG. 10 MAIN EFFECTS AND INTERACTION EFFECT PLOTS FOR FLEXURAL STRENGTH.

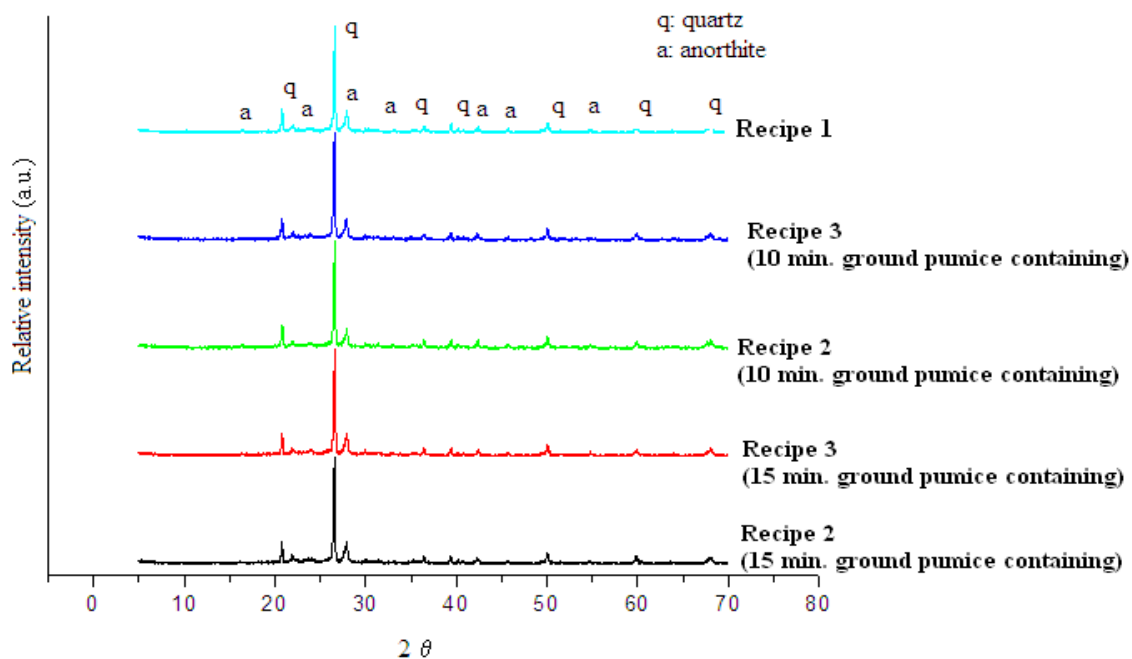


FIG. 11 XRD GRAPHS OF SAMPLES FIRED AT 1150°C.

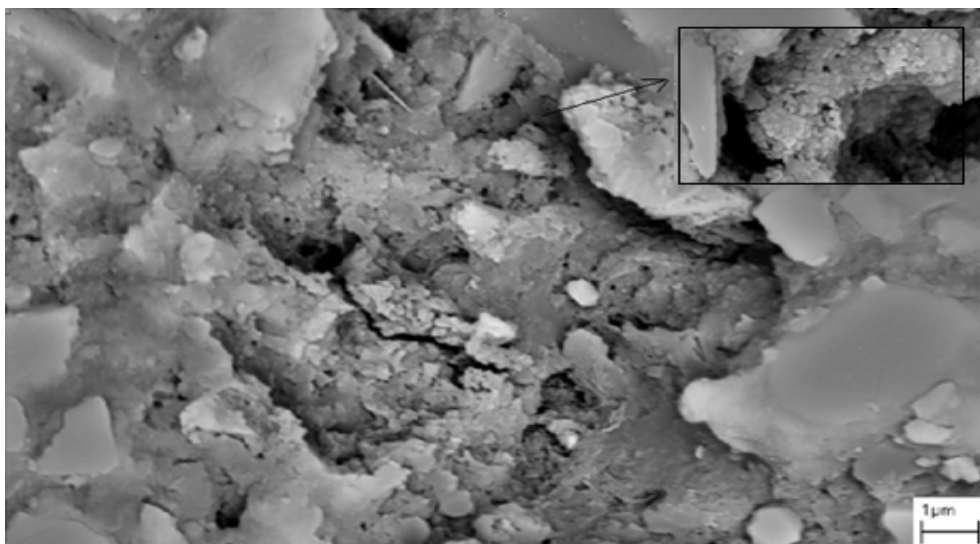


FIG. 12 A REPRESENTATIVE SE IMAGE TAKEN FROM THE POLISHED AND ETCHED SURFACE OF RECIPE 1 FIRED AT 1150°C.

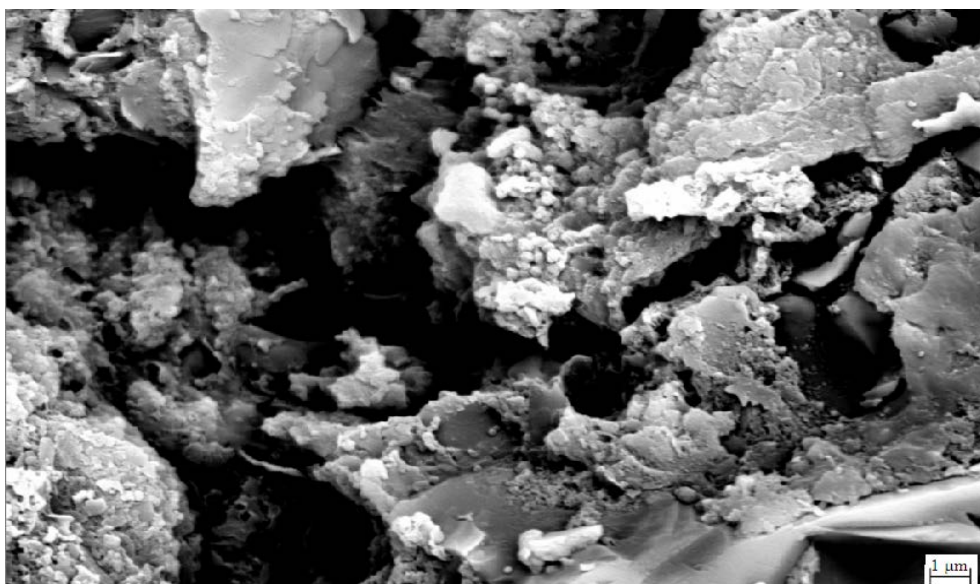


FIG. 13 A REPRESENTATIVE SE IMAGE TAKEN FROM THE POLISHED AND ETCHED SURFACE OF 10 MIN. GROUND PUMICE CONTAINING RECIPE 2 FIRED AT 1150 °C.

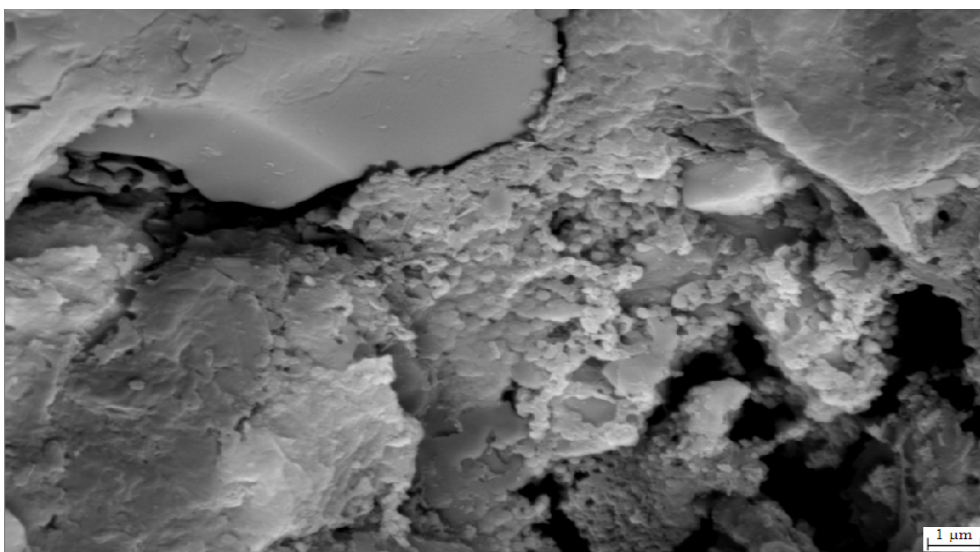


FIG. 14 A REPRESENTATIVE SE IMAGE TAKEN FROM THE POLISHED AND ETCHED SURFACE OF 10 MIN. GROUND PUMICE CONTAINING RECIPE 3 FIRED AT 1150 °C.

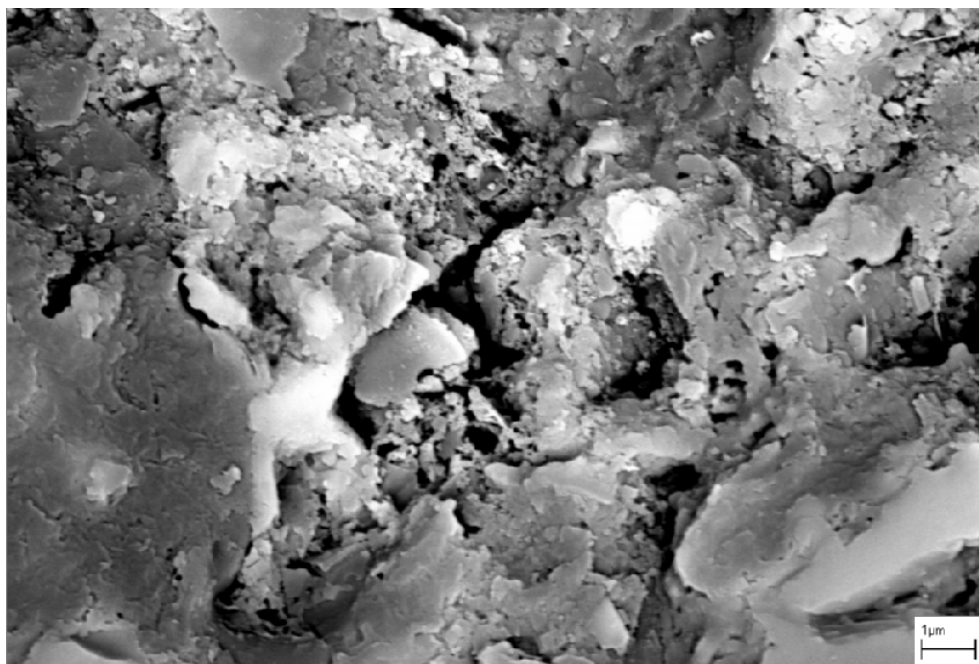


FIG. 15 A REPRESENTATIVE SE IMAGE TAKEN FROM THE POLISHED AND ETCHED SURFACE OF 15 MIN. GROUND PUMICE CONTAINING RECIPE 2 FIRED AT 1150 °C.

TABLE 6 QUANTATIVE ANALYSIS RESULTS OF SAMPLES FIRED AT 1150°C

Samples	Quartz	Anorthite	Glassy phase
Recipe 1 (0 wt. % pumice)	43.48±0.46	30.60±0.85	25.90±0.71
Recipe 2 (15 min. ground pumice containing)	42.23±0.44	26.20±0.81	31.56±0.82
Recipe 3 (15 min. ground pumice containing)	39.63±0.40	24.96±0.80	35.39±0.84
Recipe 2 (10 min. ground pumice containing)	43.53±0.48	24.17±0.85	31.97±0.80
Recipe 3 (10 min. ground pumice containing)	40.48±0.46	25.84±0.83	33.66±0.88

The pumice grinding time has a 35.5% effect on water absorption. Water absorption decreases with an increase in the amount of pumice and firing temperature, whereas it increases with an increase of milling time (Fig. 7). Increasing the amount of pumice in the samples because of the high alkali content of the pumice and increasing the firing temperature, affects the behavior of the vitrification and is thought to reduce water absorption.

For bulk density, a pumice grinding time of 26% is effective. The interaction graphs of effective factors are given in Fig. 8 on bulk density. Level 1 of pumice grinding time (10 minutes), levels 2 of the amount of pumice (7 wt.%) and firing temperature (1150 °C) increase the bulk density values. Besides the interaction factor grinding time of pumice*the amount of pumice, other interaction factors are not effective on bulk density.

The grinding time of pumice*the amount of pumice has a 43.1% effect on total porosity. The amount of the total porosity decreases with an increase in the

amount of pumice and firing temperature, but it increases with an increase in the pumice grinding time (Fig. 9).

A firing temperature of 62.5% is effective on flexural strength. In Fig. 10, it is observed that flexural strength increases with an increase of firing temperature, and that pumice grinding time factor does not make a significant impact on flexural strength. An addition of pumice to the wall tile body reduces the flexural strength values.

It is thought that the flexural strength of the wall tiles changes in relation to the amount of the phases. For this reason, the results of an XRD analysis of the samples sintered in the same firing conditions were investigated in comparison with the Rietveld method. The crystal phases obtained from the specimens are quartz and anorthite (Fig. 11). As a result of the Rietveld analysis, with an increase in grinding time and amount of pumice, the amount of the glassy phase increases and that of the anorthite crystals decreases (Table 6). With an increase in the amount of pumice

especially, the effect of an increase in total alkali oxides dissolves the quartz and anorthite crystals and leads to an increase in the amount of the glassy phase. Capoglu and Taskiran (2004), determined that anorthite based bodies reach higher flexural strength values than the commercial (standard) products. The decrease in flexural strength is thought to be due to a decrease in the amount of anorthite [Capoglu and Taskiran, 2004].

Micrographs of samples with different pumice contents, grinding times and firing temperatures are shown in Figs. 12-15. For the sample of recipe 1 (0 wt. % pumice), the amount of anorthite crystals is greater than that of the other samples. The anorthite crystals homogeneously dispersed in recipe 1, whereas they were localized in porosity regions. When recipes 2 and 3 are compared, it can be observed that with an increase in grinding time the amount of anorthite crystals also increases. Compared to recipe 1, with an increase in the total alkali content, the other samples' quartz crystals are small sized and less in quantity.

Conclusions

The study demonstrates that firing temperature has a significant effect on linear shrinkage and flexural strength, whereas the grinding time of the pumice and the amount of pumice have a significant effect on water absorption, bulk density, and the total porosity of the samples prepared with $2^2 \times 3^1$ mixed-level factorial design. Due to the high alkali content of pumice, the glassy phase of wall tiles increases and therefore, the bulk densities of the samples are higher and the total porosity of the samples are lower than that of the pumice free sample (recipe 1). The high amount of the glassy phase formation with the high total alkali oxides in the samples has enhanced the degree of densification and a low amount of porosity has been obtained.

Anorthite crystals are active on the flexural strength and in our study the dissolution of anorthite crystals is observed by the effect of the increased glassy phase resulting in a reduction in the flexural strength values. The grinding time and the amount of pumice were found to be active on examined features as well as the main factor and interaction factors. It is observed that the experimental design method is an important method for determining the effect of the gradual addition of pumice instead of pegmatite on the properties of wall tiles and how a temperature difference of even 50°C (from 1100°C to 1150°C)

changes the linear shrinkage and flexural strength values (in a short and cost-effective time frame).

ACKNOWLEDGMENT

The authors would like to thank Ceramic Research Centre Inc. (Eskisehir/Turkey) for supporting this work and also Denge Bims Pumice Plant (Nevsehir/Turkey) for providing the pumice.

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